Standard Oil Company (Indiana)

INFORMATION DIVISION TRANSLATION T46-94

API-TON Reel 71, Frames 482-485
C.I.O.S. Target 30/5.05- Rheinpreussen-Homberg. Part III
Report No. 6, by Paul Garbe, Frankfort, July 2, 1938
Concerning: Gas Desulfurization by Activated Carbon Disposal Between Coarse and Fine Purification in Rheinprussen.

After completion of experiment No. 16 with coke gas finished by Mr. Kleeman, Rheinpreussen carried out 5 further experiments with higher gas throughput - 205, 149.7, 198.4, 95.2 and 129.5 normal cubic meters (balance diagrams of all experiments are in Appendix 2). The diagram of experiment 17 shows that after a gas throughput of 150 normal cu.meters through the carbon (1.28 kg.) there occurs an increase of the sulfur content from 0.1 to 0.5 or 1.4 grams of sulfur per 100 cu.meters after the fine purification mass subsequently inserted. Sulfur content of the gases in a parallel experiment after the fine purification without carbon preceeding installations is 7 to 10 grams of sulfur per 100 cu.meters.

On basis of a suggestion by us Rheinpreussen added the exhausted benzol of the coke gas experiment #16 to the synthesis gas which has been purified with activated carbon during the whole duration of experiment 19. The benzol concentration was to amount to 4 to 5 grams per cu.meter, that is, it was to correspond to that of the coke gas. According to the determinations which had been made in the coke gas experiment (very poor sulfur removal) the sulfur compounds which were brought to the synthesis gas with the exhaust benzol were supposed to break through behind the activated carbon practically without residue, after the subsequently-placed purification mass.

Results

Average sulfur content of the activated carbon waste gases (198.4 normal cu.meters) = 9.92 grams of sulfur per 100 cu.meters.

The average sulfur amount which was added by the exhaust benzol was 2.18 grams of sulfur per 100 cu.meters.

Total sulfur content = 12.12 grams of sulfur per 100 cu.meters.

Benzol concentrations of the gas (should equal 3.7 grams per cu.meters) equals 1.9 grams/cu.meters.

The average sulfur content after the fine purification amounted to 3.02 grams of sulfur per 100 cu.meters. Since the sulfur amount with that which was carried by the exhaust benzol amounted only to 2.18 grams of sulfur per 100 cu.meters, one can assume that it breaks through completely through the fine purification.

The difference between 2.18 grams of sulfur per 100 cu.meters to 3.82 cannot be explained alone by the poor operation of the fine purification mass which was to be expected through the addition of benzol, but also through the gradually occurring using-up of the mass. Thus, for example, the average sulfur content of the gas after fine purification with activated carbon installation (without benzol admixture) amounted to 0.65 grams/100 cu.meters at the previous experiment #18 (gas throughput 149.7 normal cu.meters).

The fine purification mass was dismantled after experiment 19 and substituted by a fresh one (146 grams per pipe). The sulfur load of the substance was calculated without carbon fore- installation at 7.9%, with carbon fore installation 6.6%. Analytically a sulfur accumulation of 8.7% respectively 7.89% was determined.

After the loading number 21 the experiments were continued by us.

According to the present experimental results of the 21 loading experiments the following points were to be clarified after discussion with Dr. Grimme.

1. First, it was to be clarified whether a decomposition occurs in the benzol (about 0.5 grams per cu.meter) which is contained in the gas during fine purification, because in the desulfurising experiments which are running at present in a 3-absorber plant with synthesis gas (1000 cu. meters throughput per hour) after the fine purification no hydrocarbon condensate is recovered.

2. A carrying out of a high temperature evaporation to determine whether a greater effect than before is to be expected after increased sulfur load.

3. Determination of the action of a "benzol evaporation extraction" through addition of benzol or benzol vapor into the absorber top before the evaporation.

4. Observation of the pre-purification action of the carbon after increase of the stream velocity from 10 to 20 centimeters per second.

5. Current running of the resin test sample after the carbon- and fine-purification. Observation whether there exists a relationship between the positive result of the resin test sample after the carbon and the rise of the sulfur content after the fine purification.

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6. Controlling of the desorption of the sulfur compounds which are absorbed by the carbon at higher gas throughput (up to 200 normal cu. meters), which becomes apparent from the diagrams of experiments #17, 19 and 21.

Results:

To 1. The loading experiment #22 which was carried out with synthesis gas after fine purification decomposed the same amount of benzol (0.63 grams/cu.meters) as the experiment 23 which followed with synthesis gas before the fine purification (0.72 grams/cu.meter). The benzol losses in the activated carbon installation can be traced to errors of a technical nature (separator) which were eliminated since then.

To 2. The high temperature evaporation which was carried out after loading #23 did not bring a more favorable result than the three previous ones. The sulfur residue load could only be reduced from 1.53 to 1.43%.

To 3. After each loading #24 and 25, a "benzol evaporation extraction" was carried out. In the first case 401 grams of thiophene-free benzol was introduced as liquid in the absorber top and subsequently evaporated. In the second case 402 grams of thiophene-free benzol was distilled over into 100°C hot carbon, then the carbon was further cooled and finally evaporated. In neither case could even the sulfur amount which was taken up by the carbon during the loading be driven off.

To 4. Experiment 25 which was carried out with a flowing velocity of 20 cc per second and increased gas throughput (230 cu.meters) shows no difference in comparison with experiments carried out with less flowing velocity (10 cc per second). That is, the desulfurizing performance of the carbon amounted to an average of about 20%. The rise of the sulfur content after the fine purification occurs only after a gas throughput of 190 normal cu.meters. The rise is from 0.2 to 0.37 grams of sulfur/100 cu. meters.

To 5. Here it could be determined without doubt during experiment 25 that there exists a parallel between the positive result of the resin test sample and the rise of the sulfur content after the fine purification. (see diagram)

To 6. In experiment 25 no desorption of the sulfur compounds which were absorbed by the carbon during increased gas throughput could be determined. The desulfurizing of the carbon at the end of the loading amounted still to 27%, or 11% after subtracting the inorganic sulfur which was contained in the charged gas.

Dr. Grimme will finish experiment 26 with particular regard for point 5 and then will break off the experiments. Until that time about 1,500 normal cu.meters of gas will pass through per kg. of carbon.

/s/ P. Garbe

(In the original, frames 486-515, follow tables and diagrams of experiments 17-25.

Translated Oct. 30, 1946 - Rochelle H. Bondy
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